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FRACTIONATION OF SOYBEAN OIL BY LIQUID-LIQUID EXTRACTION 1/

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For some time we have been working at this Laboratory on a process for separating soybean oil into two fractions by an operation known as liquid-liquid extraction. The outlets for soybean oil are numerous, but they can, in general, be grouped into two classes, an outlet for food purposes and one for industrial uses. The desirable characteristics of an oil for either outlet differ considerably from those preferred for the other. An edible fat should be bland in taste, resistant to changes in flavor, light in color, and have non-drying characteristics. A drying oil, such as linseed oil for paints and varnishes, should dry to form a hard, tough film in a reasonable length of time. An excellent edible oil is cottonseed oil; it is classified as non-drying. The oil from soybeans, in respect to these classes, falls midway between cottonseed and linseed oils, and usually is classified as semi-drying.

Soybean oil is a complex mixture of chemical compounds. Since it is used in the manufacture of a variety of products ranging from margarine to linoleum, it would seem desirable to separate the oil into two or more fractions, each of which we know is better suited for a specific purpose than the parent oil. This is what we attempt to do in our liquid-liquid extraction process, which is sometimes referred to also as solvent fractionation, or selective extraction. Our aim is to produce from soybean oil a fraction which will be excellent for use in paints and another that can be manufactured into edible products.

As an illustration of how this can be done, suppose we mix one volume of soybean oil with nine volumes of a solvent, such as furfural, then put the mixture in a bottle and shake it gently. After a few minutes, the mixture will form two layers. The furfural has dissolved part of the oil, but not all. The solvent is about one-fourth heavier than the oil. Therefore, the bottom layer will consist of furfural with some oil in solution. The remainder of the oil is in the top layer and some furfural will be mixed with it. If this top layer is removed and the furfural evaporated off, we shall find that this oil has an iodine value of 126, as compared with 133 for the original oil. Likewise, by removing the furfural from the bottom layer, analysis of its oil will give an iodine value of 139. Therefore, by extracting the oil with furfural, we

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2/ One of the Laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U.S. Department of Agriculture

have produced two fractions whose iodine values differ by 13 units.

The "iodine value" results from a test made on an oil to determine its chemical characteristics. It is indicative of the oil's ability to dry into a hard film and also helps to classify the oil for edible purposes. Cottonseed oil, which is used in the manufacture of shortening and margarine, has an iodine value of approximately 105. On the other hand, linseed oil, which is in much demand for the production of paints and varnishes, has an iodine value of 170 or more.

We have shown that furfural is a selective solvent, because it will dissolve only part of the soybean oil and that the part which is dissolved differs in its characteristics both from the original oil and from the part that did not go into solution. Furfural is made from corncobs, oat hulls, cottonseed hulls, and similar agricultural residues.

The next problem is to increase the fractionation or the difference in iodine values of the products. We usually refer to this difference as the iodine value "spread." If several bottles were arranged in series, oil fed in at one end of the group and furfural at the other, and the two layers which are formed in the bottles separated and passed in opposite directions, we would increase the fractionation. This has been done on a commercial scale in the petroleum industry for refining lubricating oils by the use of a series of mixers and settlers. Later, it was found that a vertical tower filled with special packing would give results equally as good, and in many cases better, while it also made the process easier to operate.

A cross-sectional view of our extraction column is shown in Figure 1. It consists of a vertical tube 2.5 inches in diameter, about 18 feet high and filled with small porcelain rings which are  $1/4$  inch in diameter and  $1/4$  inch long. A 4-inch pipe surrounds the tube for a distance of about 14 feet. Hot water is circulated through this jacket to maintain the column at the desired temperature. Since part of the tube was not jacketed originally, we have installed a water bath at the bottom and built a box around the mid-section. Heated air is blown through this box. By these means, we can control the temperature within the column for its entire length.

Three streams enter the tower or column and two leave it. Since furfural has the greatest density, it is fed in at the top. Oil is introduced at a midpoint, and a second solvent, naphtha, is pumped in at the bottom. This naphtha is comparatively light and rises towards the top, counter-current to the flow of the furfural. Before the column is put into operation, the lower half is filled with furfural and the upper half with oil. The boundary between these two layers is known as the interface and its location is observed by means of a gauge glass.

During normal operation of the tower, the furfural trickles down over the packing to the lower end, dissolving some of the soybean oil during



the course of its passage. As this mixture of furfural and oil reaches the bottom of the tower, it meets the naphtha, which washes part, but not all of the oil away from the furfural. Since furfural is a selective solvent, it will retain the oil having the highest iodine value. Consequently, we obtain an improved industrial oil by this means. The solution emerging from the column at the bottom is known as the extract phase. It consists of oil and naphtha dissolved in furfural.

That portion of the oil not carried out with the furfural rises through the packing and flows out of the column at the top. Of course, during its passage, a continual interchange occurs between the furfural and the oil. This entire operation is described as continuous counter-current extraction with reflux. The naphtha is the reflux solvent.

The next step is the removal of the solvent from these two oil fractions. This is conducted in several evaporators and stripping stills of conventional design. In Figure 2 we have a schematic arrangement for the entire extraction process. Essentially, it consists of three steps: first, extraction of the oil; second, removal of the solvent from each fraction; and third, purification of the solvent. It will be noted that the extraction column is a small part of all of the equipment used. The solvents are recycled continuously, and two fractions, the raffinate product and the extract product, are obtained from the feed oil.

Naturally, we have been interested in learning the qualities of these two fractions and determining how they compare with the original soybean oil. Mr. Lewis of the Oil and Protein Division has made paints from a series of our extract oils and determined their drying rates. The results are given in Table 1 below:

Table 1.--Drying rates of paints

<u>Oil vehicle</u>	<u>Iodine value</u>	<u>Drying rate hours</u>
Original soybean	134	9.5
Extract oil	144	8
"	151	7.8
"	155	6.5
"	161	6
"	165	6
"	168	5.8
Linseed	184	4

The paint made from the feed oil having an iodine value of 134 dried in 9.5 hours. The paints made from the extract fractions with iodine values ranging from 144 to 168 required from 8 to 5.8 hours to produce more or less tack-free films. In comparison, the drying rate for linseed oil was 4 hours. Thus, we see that the drying time decreases as the iodine value increases, and all of the extract oils showed an

improvement over the degummed soybean oil.

Dr. Dutton's group has been investigating the flavor stability of the raffinate oil. Its stability appears to be about equal to that of the original oil, but not much better. We may find it necessary to replace some of the antioxidants, since their concentration in the raffinate oil apparently is reduced during the fractionation process.

The distribution of several other minor constituents is shown in Table 2:

Table 2.--Distribution of minor constituents of soybean oil between extract and raffinate phases

Constituent	Oil		
	: Original	: Extract	: Raffinate
Break material, percent	0.11	0.06	0.18
Phosphorus, percent	0.011	0.0003	0.019
Unsaponifiable matter, percent	0.58	0.61	0.36
Free fatty acids, percent	0.28	0.18	0.09
Iodine value	135.4	154.4	108.7
Yield, percent	100	58	42

Most of the break material and the phosphatides are concentrated in the raffinate fraction, while the free fatty acids and the unsaponifiable matter appear in greater proportions in the extract oil. Normally, more fatty acids will be found in the extract fraction, but during these experiments all the acids were not stripped from the furfural before the solvent was redistilled.

One feature of the liquid-liquid extraction process is the possibility it offers for varying the ratio of the product oils. By adjusting the rate at which naphtha is fed to the extraction column, the extract yield can be varied from 20 to 80 percent of the original oil. Of course, the iodine values of the two fractions change accordingly. Figure 3 illustrates this very clearly. In this series of experiments we obtained yields of extract oil ranging from 23 to 83 percent, with the iodine values decreasing meanwhile from 170 to 144. Those for the raffinate fraction began at 122 and diminished progressively to 95. As the quantity of either fraction is increased, its iodine value approaches that of the feed stock.

A comparison made of the Lovibond colors for the three oils at various stages in the conventional refining process used for producing an edible fat showed interesting responses for each. This is illustrated in Figure 4. Both the extract and raffinate fractions were lighter in color than the original oil. After each was refined, bleached, and deodorized, the raffinate oil was much lighter than either of the other two, and

the extract fraction was slightly better than the original oil.

In the development of a satisfactory process such as this, a number of variables must be investigated. Among them are solvent ratios, type of packing to be used in the column, height of packing required, capacity of the column, location of the feed inlet, design of the settling zones within the column, extraction temperature, and effect of various feed stocks upon the characteristics of the products. The object of the pilot-plant work is to study these variables in small-scale equipment of the type that would be used for commercial operations and to determine the probably cost of conducting the process on a large scale.

The fractionation obtained for various solvent ratios varies considerably. The data in Table 3 illustrate the benefits obtained by the use of more solvent.

Table 3.--Effect of solvent ratio

<u>Ratio of solvent to oil</u>	<u>Iodine value "spread"</u>
20	41.1
13	38.3
10	33.2
5	24.1

At a ratio of 20 to 1, the iodine value "spread" was 41.1, but as the ratio was decreased to 5, the spread fell off to 24.1 accordingly. Of course, the cost of operating the process is increased by use of the higher ratios.

The effect of mass velocity was not as great as expected. The data for several experiments are listed in Table 4.

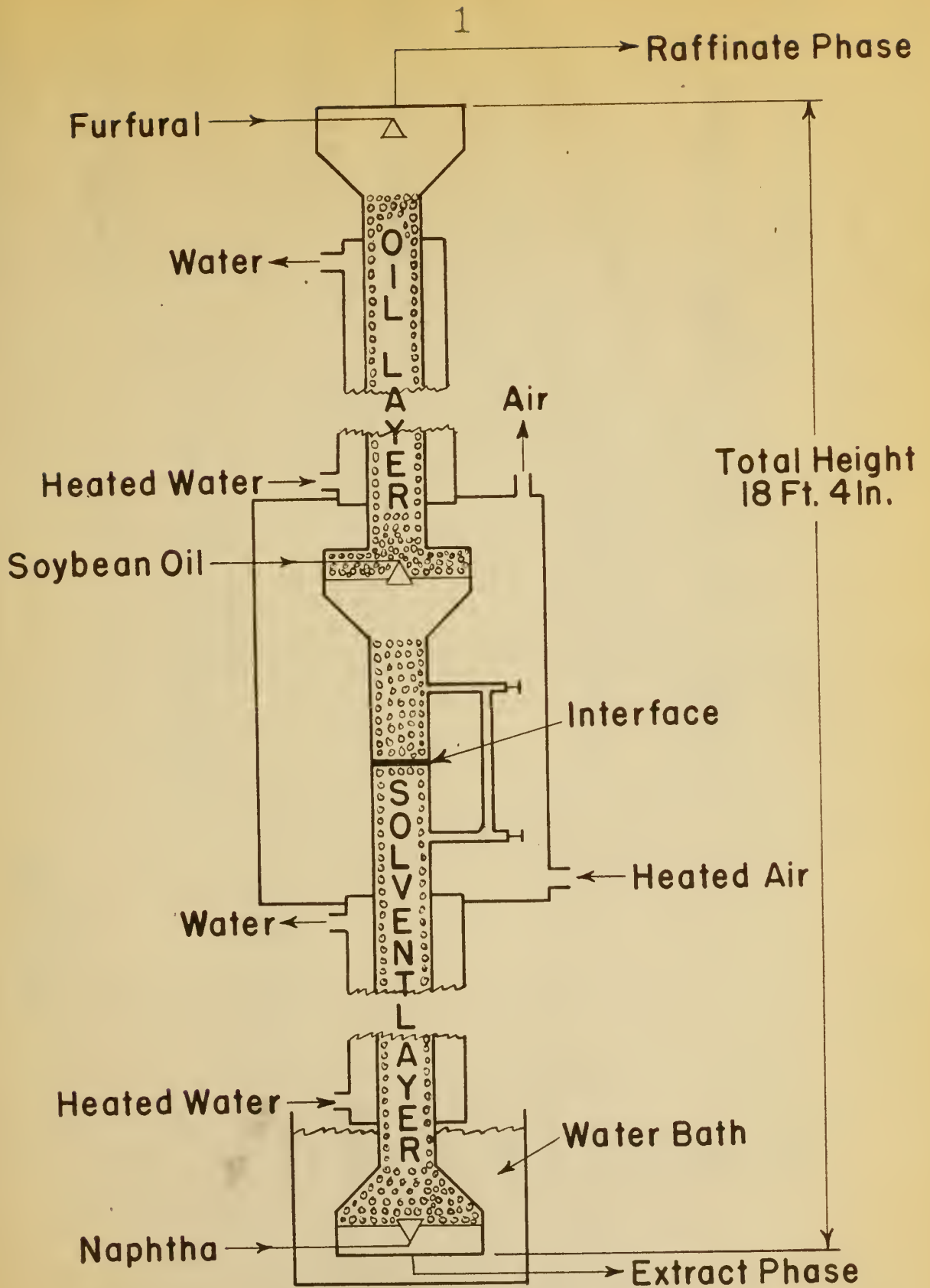
Table 4.--Effect of mass velocity

<u>Oil feed rate, lb./hr./sq. ft.</u>	<u>Iodine value spread</u>
62	38.1
92	37.1
122	36.5
152	Flooding

Most of our tests have been made at an oil rate of 62 pounds per hour per square foot of cross-sectional area in the tower. When the capacity has been increased by approximately 200 percent, the iodine value "spread" has fallen off only 1.6 units. However, when we have attempted to raise the oil input still further, the column has not functioned properly and poor fractionation has been obtained.









# FRACTIONATION OF SOYBEAN OIL

